Thermal analysis evaluation of mechanical properties changes promoted by gamma radiation on surgical polymeric textiles

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Abstract

The large number of surgical operations with post-operative infection problems and the appearing of new infectious diseases, contribute to the development of new materials in order to answer the needs of health care services. This development must take into account the modifications promoted by sterilisation methods in materials, namely by gamma radiation. The differential scanning calorimetry (DSC) and thermogravimetry (TGA) techniques show that a nonwoven and a laminate textiles maintain a good molecular cohesion, do not showing high levels of degradation, for gamma radiation dose values lower than 100 kGy in nonwoven and 200 kGy in laminate materials. The tensile strength and the elongation decrease slowly for the nonwoven textile and decrease faster for the laminate textile for 25 and 80 kGy absorbed dose. This paper shows that the DSC and TGA techniques can be helpful for the prevision of mechanical changes occurred in the materials as a consequence of the gamma irradiation. © 2002 Elsevier Science B.V. All rights reserved.

Keywords: Polymeric surgical textiles; Thermal analysis; MCNP; Radiosterilisation

1. Introduction

Nowadays, the large number of surgical operations with post-operative infection problems (in Europe, in average between 5% and 10%) and its tendency to increase, as well as the appearing of new infectious-contagious diseases, contribute to accelerate the development of new materials in order to answer the requirements and specifications of health care services.

Although, the selection of the most appropriate surgical protection is very complex as a consequence of the different parameters that have to be conjugated. Surgical exigencies, type of material and their related properties (mechanical, barrier, comfort, etc.) and the changes promoted by the lethal agent in the materials during the sterilisation process, are parameters that must be taken into account.

Some polymeric based textiles, mainly nonwovens and laminates, are showing to have the...
adequate properties, essentials for the manufac-
turing of disposable surgical protection clothing [1].
Ionising radiation (gamma and electron beam
radiation) is often used for the sterilisation of
polymeric based disposable medical devices.
Nevertheless this sterilisation can induce some
changes in their behaviour and some of them could
restrict their use for the defined applications. The
mechanical properties (tensile strength and elonga-
tion) are commonly regarded as a rule of quality
and they must be evaluated before and after ste-
rilisation treatment. These data should be included
in the assessment of the quality of a textile material.
This work is part of a study of the influence of
ionising radiation (gamma and e-beam radiation)
on the properties and performance of disposable
materials and products for hospitalar protection.
In this paper will be presented the results obtained
with two textiles, a nonwoven and a laminate, ir-
niated with gamma radiation at the Co-60 Por-
tuguese Gamma Facility (UTR) [2].
According to the standards [3], this kind of re-
search requires the irradiation of lots of samples to
a maximum acceptable dose for the products.
Knowing materials behaviour profile with ra-
diation, in what concerns to a few properties di-
rectly dependent of the molecular organisation, it
is possible to foresee what will happen with other
essential properties. This is the case of melting
temperature (T_m), melting enthalpy (ΔH_m) and the
temperature at which the materials begin to de-
grade (T_deg). These properties can be measured by
thermal analysis techniques (e.g. differential scan-
ning calorimetry (DSC) and thermogravimetry
(TGA)) [4].
Both, DSC and TGA techniques, require only
few milligrams of material to perform a thermo-
gram. Then, instead of irradiate a lot of samples in
their final form, it is possible to irradiated little
samples of material at several doses. These pro-
cedures allow a quick knowledge of material be-
haviour and the identification of the critical doses.

2. Experimental details

FAPOMED SA, Felgueiras, Portugal processed
textile samples of nonwoven and laminate mate-
rials. These textiles have the following composition
and density:
Nonwoven: (55% cellulose, 45% polyester); \( \rho = 0.17 \).
Laminate: 1st layer: 0.02 kg low density poly-
ethylene (LDPE) film; 2nd layer: nonwoven (70%
vicose, 30% polyester); \( \rho = 0.14 \).
Eight samples (0.1 x 0.1 m²) of each material
were packed in a proper bag, evacuated and
sealed.
Two batches of 25 gowns manufactured from
each material were packed in the same conditions
and were accommodated in pasteboard boxes of
0.4 x 0.4 x 0.4 m³ (standard boxes of UTR).
The samples were irradiated at the UTR. The
small ones were irradiated in very well charac-
tised steady positions, with doses of 25, 80, 150,
200, 250, 300, 350 and 400 kGy.
The big samples (gowns) were irradiated in the
pasteboard boxes in dynamic process for a final
dose of 25 and 80 kGy.
The irradiations control parameters were mon-
itorised with PMMA dosimeters (Amber Perspex
[1, 30 kGy] and Red Perspex [1, 50 kGy]) [5], be-
ing the dose values calculated by Monte Carlo
N-Particle Transport Code (MCNP code) [6–8],
which allows the planning of the irradiation.
The small samples irradiated with doses between
25 kGy and 400 kGy and a nonirradiated one
(blank) were analysed by DSC and TGA tech-
niques. These assays were performed with equip-
ment from the TA Instruments, at the Polymer
Characterisation Laboratory of ITN, Sacavém,
Portugal.
DSC and TGA thermograms were obtained
under the same experimental conditions: nitrogen
flux of 0.01 l/s and with standard heating programs
for polymer analysis.
Concerning the big samples, the textiles me-
chanical properties were measured and statistically
analysed at UM – Department of Textile Engi-
neering, Guimarães, Portugal [9], before and after
irradiation at 25 and 80 kGy.
For the mechanical tests it was used a pneu-
matic dynamometer from Hounsfield Equipment
(charge cell: 20–1000 N). The tests were realised in
agreement with the standard guidelines for medical
textiles [3,10].
3. Results and discussion

For the steady and dynamic irradiations the previsions of dose performed by MCNP code show a maximum difference of 2%. The dose uniformity for these irradiations was $U(25 \text{ kGy}) = 1.11$ and $U(80 \text{ kGy}) = 1.18$.

Figs. 1 and 2 show the variation of melting enthalpy ($\Delta H_m$) and melting temperature ($T_m$) with the absorbed gamma radiation dose, for nonwoven and laminate textiles. Fig. 3 shows the variation of temperature of beginning of degradation ($T_{deg}$), of nonwoven and laminate textiles, with the absorbed gamma radiation dose.

For evaluation of the relevant mechanical properties purpose, in different irradiation states and fibre material orientation (MD – machine direction; CD – cross direction of the material) 20 measurements of each material were performed. Nonwoven and laminate materials were manufactured from the same conditions of UTR. The same conditions were used to vacuum evacuate and irradiate in the same conditions for a final process for a final evacuation.

The statistical study concerned the test of variance (F-Snedecor) and the test of the means ($t$-Student-Fisher), with 0.05 level of significance to both distributions.

Figs. 1 and 2 suggest that the materials maintain a good molecular cohesion in the range of dose values considered. Fig. 3 do not show the existence in the materials of high levels of degradation, for doses values lower than 100 kGy for nonwoven and 200 kGy for laminate material.

Nonwoven is a cellulose based material. As it is known, cellulose is a preferential degrading material under gamma influence [11]. Being cellulose the backbone of the textile is expected to see a rapid degradation of the material with the increasing dose (see Fig. 3). Nevertheless, polyester is a preferential cross-linking material [11]. The
Table 1

Effects on the tensile strength and elongation of nonwoven material, with a dose of 25 and 80 kGy (fibre direction dependence)

<table>
<thead>
<tr>
<th>Gamma radiation dose</th>
<th>Mechanical property (average values)</th>
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<td></td>
<td>$F_{\text{max}}$ (N)</td>
<td>$E_{\text{max}}$ (%)</td>
<td>$F_{\text{break}}$ (N)</td>
<td>$E_{\text{break}}$ (%)</td>
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<td>83.95</td>
<td>131.88</td>
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<tr>
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<td>81.35</td>
<td>130.80</td>
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<td>1.03</td>
<td>1.27</td>
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</table>

S: Significant; NS: Non-significant.

Table 2

Effects on the tensile strength and elongation of laminate material, with a dose of 25 and 80 kGy (fibre direction dependence)

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<td>$E_{\text{max}}$ (%)</td>
<td>$F_{\text{break}}$ (N)</td>
<td>$E_{\text{break}}$ (%)</td>
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<tr>
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<td>$t$</td>
<td>8.13</td>
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increase of reticulation promoted in polyester acts in the sense of the increase of mechanical resistance and the degradation occurred in cellulose acts in opposite sense. So, under gamma irradiation, will be expected that the internal mechanical resistance of the textile does not vary substantially (see Fig. 1).

In what concerns with tensile strength and elongation is expected that the tensile strength will decrease slowly as dose increase, as so the elongation (depending on the fibres orientation), as can be observed in Table 1. In effect, according to these data the maximum variation for tensile strength is 10% (for $F_{\text{break}}$, MD direction and 80 kGy conditions) and 3% for the elongation (for $E_{\text{break}}$, CD direction and 25 kGy conditions).

Laminate is a double layer textile. Both layers have preferential cross-linking polymers in their composition: LDPE in the first layer and polyester in the second. Viscose is a cellulose derivative and is a preferential degrading polymer [11]. The behavior of the second layer under gamma irradiation is expected to be similar to the nonwoven cellulose based textile under the same conditions. But in the laminate textile, the first layer will increase the initial resistance of the material as a
result of the LDPE reticulation. So, the viscose degradation effects are opposite to the polyester and LDPE reticulation effects. Due to this the textile will suffer a small improvement of its thermal resistance to a dose near 200 kGy, from where LDPE and polyester begin to degrade too (see Fig. 3). After that dose value, it is expected the loose of textile internal mechanical resistance (see Figs. 2 and 3).

In the case of tensile strength and elongation is expected that tensile strength will decrease as dose is increasing (depending on the 2nd layer fibres orientation) and the elongation will be more seriously reduced as it was in nonwoven.

In effect, according to the data shown in Table 2 the maximum variation for tensile strength is 22% (for $F_{\text{break}}$, MD direction and 80 kGy conditions) and 36% for the elongation (for $E_{\text{max}}$, MD direction and 80 kGy conditions).

Even that available data suggest that both textiles maintain their mechanical and thermal stability in the range of dose values usually applied to medical devices sterilisation (15–40 kGy), this study must be complemented with data from other essential materials properties (e.g. barrier properties).

### 4. Conclusion

Gamma radiation can promote changes in the molecular structure of the materials, leading to an improvement or degradation of their properties. Thermal techniques as DSC and TGA, may be used for evaluation of some properties that are directly dependent of materials molecular organisation. In this way it is possible to use thermal analysis to predict the behaviour of other essential properties, for instance mechanical properties changes occurred in studied textiles due the gamma irradiation.

### Acknowledgements

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### References


[3] EN 552 Sterilisation of Medical Devices Validation and Routine Control of Sterilisation by Irradiation.


